

We claim:

1. A polymorphic form of 9-nitrocamptothecin, the polymorph being characterizable as having, by differential scanning calorimetry, no observable endotherm and an exotherm at between 273.6 and 275.6 °C, and a solution NMR spectrum with multiplets at 1.7 and 3.7 ppm shifts.
2. A polymorphic form of 9-nitrocamptothecin according to claim 1, the polymorph being further characterizable as having an exotherm by differential scanning calorimetry at between 274.1 and 275.1 °C.
3. A polymorphic form of 9-nitrocamptothecin according to claim 1, the polymorph being further characterizable as having an exotherm by differential scanning calorimetry at between 274.4 and 274.8 °C.
4. A polymorphic form of 9-nitrocamptothecin according to claim 1, the polymorph being further characterizable as having an exotherm by differential scanning calorimetry at between 274.5 and 274.7 °C.
5. A polymorphic form of 9-nitrocamptothecin according to claim 1, wherein the polymorph is obtained by grinding.
6. A polymorphic form of 9-nitrocamptothecin, the polymorph being characterizable as having an X-ray powder diffraction pattern with diffraction lines at  $2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of wavelength 1.5406 Angstrom.

1 7. A polymorphic form of 9-nitrocamptothecin, the polymorph being  
2 characterizable as having an X-ray powder diffraction pattern with diffraction lines at  
3  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of wavelength 1.5406  
4 Angstrom.

1 8. A polymorphic form of 9-nitrocamptothecin, the polymorph being  
2 characterizable as having, for Cu  $K\alpha$  radiation of wavelength 1.5406 Angstrom, an X-  
3 ray powder diffraction pattern with diffraction lines at  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and  
4 23.9.

1 9. 9-nitrocamptothecin in a form crystallized from tetrahydrofuran.

1 10. A polymorphic form of 9-nitrocamptothecin according to claim 10, the  
2 polymorph being characterizable as having, by differential scanning calorimetry, no  
3 observable endotherm and an exotherm at between 273.6 and 275.6  $^{\circ}\text{C}$ , and a solution  
4 NMR spectrum with multiplets at 1.7 and 3.7 ppm shifts.

1 11. A polymorphic form of 9-nitrocamptothecin according to claim 10, the  
2 polymorph being characterizable as having an X-ray powder diffraction pattern with  
3 diffraction lines at  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of  
4 wavelength 1.5406 Angstrom.

1 12. A polymorphic form of 9-nitrocamptothecin according to claim 10, the  
2 polymorph being characterizable as having an X-ray powder diffraction pattern with  
3 diffraction lines at  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of  
4 wavelength 1.5406 Angstrom.

1 13. A polymorphic form of 9-nitrocamptothecin according to claim 10, the  
2 polymorph being characterizable as having, for Cu  $K\alpha$  radiation of wavelength 1.5406

3 Angstrom, an X-ray powder diffraction pattern with diffraction lines at  $2\theta$  values 6.7,  
4 12.5, 14.0 and 23.9.

1 14. A pharmaceutical composition comprising:  
2 a pharmaceutical carrier; and  
3 a polymorphic form of 9-nitrocamptothecin, the polymorph being  
4 characterizable as having, by differential scanning calorimetry, no observable  
5 endotherm and an exotherm at between 273.6 and 275.6 °C, and a solution NMR  
6 spectrum with multiplets at 1.7 and 3.7 ppm shifts.

1 15. A pharmaceutical composition according to claim 14, the polymorph being  
2 further characterizable as having an exotherm by differential scanning calorimetry at  
3 between 274.1 and 275.1 °C.

1 16. A pharmaceutical composition according to claim 14, the polymorph being  
2 further characterizable as having an exotherm by differential scanning calorimetry at  
3 between 274.4 and 274.8 °C.

1 17. A pharmaceutical composition according to claim 14, the polymorph being  
2 further characterizable as having an exotherm by differential scanning calorimetry at  
3 between 274.5 and 274.7 °C.

1 18. A pharmaceutical composition comprising:  
2 a pharmaceutical carrier; and  
3 a polymorphic form of 9-nitrocamptothecin, the polymorph being  
4 characterizable as having an X-ray powder diffraction pattern with diffraction lines at  
5  $2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of wavelength 1.5406  
6 Angstrom.

1 19. A pharmaceutical composition comprising:  
2 a pharmaceutical carrier; and  
3 a polymorphic form of 9-nitrocamptothecin, the polymorph being  
4 characterizable as having an X-ray powder diffraction pattern with diffraction lines at  
5  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of wavelength 1.5406  
6 Angstrom.

1 20. A pharmaceutical composition comprising:  
2 a pharmaceutical carrier; and  
3 a polymorphic form of 9-nitrocamptothecin, the polymorph being  
4 characterizable as having, for Cu  $K\alpha$  radiation of wavelength 1.5406 Angstrom, an X-  
5 ray powder diffraction pattern with diffraction lines at  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and  
6 23.9.

1 21. A pharmaceutical composition comprising:  
2 a pharmaceutical carrier; and  
3 a polymorphic 9-nitrocamptothecin in a form crystallized from  
4 tetrahydrofuran.

1 22. A pharmaceutical composition according to claim 21, the polymorph being  
2 characterizable as having, by differential scanning calorimetry, no observable  
3 endotherm and an exotherm at between 273.6 and 275.6  $^{\circ}\text{C}$ , and a solution NMR  
4 spectrum with multiplets at 1.7 and 3.7 ppm shifts.

1 23. A pharmaceutical composition according to claim 21, the polymorph being  
2 characterizable as having an X-ray powder diffraction pattern with diffraction lines at  
3  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of wavelength 1.5406  
4 Angstrom.

24. A pharmaceutical composition according to claim 21, the polymorph being characterizable as having an X-ray powder diffraction pattern with diffraction lines at  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of wavelength 1.5406 Angstrom.

25. A pharmaceutical composition according to claim 21, the polymorph being characterizable as having, for Cu  $K\alpha$  radiation of wavelength 1.5406 Angstrom, an X-ray powder diffraction pattern with diffraction lines at  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9.

26. A method of preparing a polymorphic form of 9-nitrocamptothecin, the method comprising:  
crystallizing 9-nitrocamptothecin from tetrahydrofuran.

27. A method according to claim 26, the polymorph being characterizable as having, by differential scanning calorimetry, no observable endotherm and an exotherm at between 273.6 and 275.6  $^{\circ}\text{C}$ , and a solution NMR spectrum with multiplets at 1.7 and 3.7 ppm shifts.

28. A method according to claim 26, the polymorph being characterizable as having an X-ray powder diffraction pattern with diffraction lines at  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of wavelength 1.5406 Angstrom.

29. A method according to claim 26, the polymorph being characterizable as having an X-ray powder diffraction pattern with diffraction lines at  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9 for Cu  $K\alpha$  radiation of wavelength 1.5406 Angstrom.

30. A method according to claim 26, the polymorph being characterizable as having, for Cu  $K\alpha$  radiation of wavelength 1.5406 Angstrom, an X-ray powder diffraction pattern with diffraction lines at  $^{\circ}2\theta$  values 6.7, 12.5, 14.0 and 23.9.